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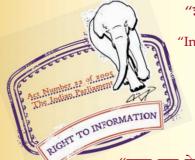
मानक

IS 13978 (1994): Zinc Diethyl dithiocarbamate (ZDEC) [PCD 13: Rubber and Rubber Products]



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## भारतीय मानक

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## Indian Standard

# ZINC DIETHYL DITHIO CARBAMATE ( ZDEC ) — SPECIFICATION

UDC 678.4.044.44

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BUREAU OF INDIAN STANDARDS MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG NEW DELHI 110002

December 1994

**Price Group 2** 

#### FOREWORD

This Indian Standard was adopted by the Bureau of Indian Standards, after the draft finalized by the Rubber Products Sectional Committee had been approved by the Petroleum, Coal and Related Products Division Council.

Zinc diethyl dithio carbamate is an ultra fast accelerator normally used as booster in combination with thiazoles and sulphenamides in dry rubber compounding. It is also a widely used curing agent in rubber latex compounding.

It is insoluble in water, alcohol and petroleum ether but partially soluble in benzene and chloroform.

As per the present state of knowledge, toxicity level of the material is low. It does not impart any taste to food products coming in contact with the rubber vulcanizate and is permitted by the German Food Law up to a total concentration of 1.2 percent and FDA (USA) up to 1.5 percent maximum on the rubber mix for rubber products coming in contact with foodstuffs.

The standard includes a recommended procedure for evaluating the material by means of a standard compounding and vulcanizing procedure.

For the purpose of deciding whether a particular requirement of this standard is complied with, the final value, observed or calculated, expressing the result of a test or analysis, shall be rounded off in accordance with IS 2:1960 'Rules for rounding off numerical values (*revised*)'. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

## Indian Standard

# ZINC DIETHYL DITHIO CARBAMATE (ZDEC) – SPECIFICATION

#### **1 SCOPE**

This standard prescribes the requirements and methods of sampling and test for zinc diethyl dithio carbamate (ZDEC) (both surface modified and unmodified) intended for use on rubber compounding as an ultra-fast accelerator or booster accelerator, both in dry rubber and rubber latex based compounds.

#### **2 NORMATIVE REFERENCES**

The following standards contain provisions which through reference in this text, constitute provisions of the standard. At the time of publication, the editions indicated were valid. All standards are subject to revision, and parties to agreements based on the standard are encouraged to investigate the possibility of applying the most recent edition of the standards indicated below:

IS No.	Title	
1070 : 1992	Reagent grade water ( third revision )	
1675 : 1971	Stearic acid, technical (first revision)	
1683:1 <b>99</b> 4	Barytes for rubber industry (second revision)	
3399 : 1993	Zinc oxide for rubber industry (second revision)	
3400 (Part 1): 1987	Methods of test for vulcanized rubbers : Part 1 Tensile stress- strain properties ( second revision )	
3400 (Part 2): 1980	Methods of test for vulcanized rubbers : Part 2 Hardness (first revision)	
3660 (Part 7): 1988	Method of test for natural rubber : Part 7 Determination of Mooney viscosity (NR : 8) (second revision)	
4588:1986	Rubber, raw, natural ( third revision )	
6918:1972	Mercaptobenzothiazole	
7086 (Part 1): 1973	Method of sampling and test for rubber compounding ingre- dients, Part 1	
8851:1994	Sulphur for rubber industry (first revision)	

#### **3 REQUIREMENT**

#### 3.1 Description

The material shall be in the form of white powder free from any visible impurities.

**3.2** The material shall comply with the requirements given in Table 1 when tested according to the procedures given in col 4 of the table.

**3.2.1** All tests shall be carried out within one month of the date of receipt of the material by the purchaser.

#### 3.3 Compound Performance Requirement

The material when compounded and tested in accordance with Annex B shall have its performance comparable with that of the sample previously approved by the purchaser.

#### **4 PACKING AND MARKING**

**4.1** The material shall be packed as agreed to between the purchaser and the supplier.

**4.2** The packages shall be securely closed and legibly marked to furnish the following information:

- a) Name of the material,
- b) Indication of the source of manufacture,
- c) Net mass of the material,
- d) Lot and batch number, and
- e) Month and year of manufacture.

**4.2.1** The package may also be marked with the Standard Mark.

4.2.2 The use of the Standard Mark is governed by the provisions of Bureau of Indian Standards Act, 1986 and the Rules and Regulations made thereunder. The details of conditions under which the licence for the use of Standard Mark may be granted to manufacturers or producers may be obtained from the Bureau of Indian Standards

#### 5 SAMPLING AND CRITERIA FOR CONFORMITY

5.1 The representative samples of the material shall be drawn as prescribed in 1S 7086 (Part 1): 1973.

#### 5.2 Number of Tests

Tests for all characteristics shall be conducted on a composite sample.

#### 5.3 Criteria for Conformity

The lot shall be considered as conforming to the specification if the composite sample satisfies each one of the requirements.

#### **6 TEST METHODS**

6.1 Test shall be conducted according to the methods prescribed in col 4 of Table 1.

#### 6.2 Quality of Reagents

Unless specified otherwise 'pure chemicals' and distilled water (see IS 1070: 1992) shall be employed in tests.

NOTE — 'Pure chemicals' shall mean chemicals that do not contain impurities which affect the results of analysis.

#### Table 1 Requirements of Zinc Diethyl Dithio Carbamate (ZDEC)

Sl No.	Characteristic	Requirement	Method of Test, Ref to Cl No.
(1)	(2)	(3)	(4)
i)	Melting point:		
	<ul><li>a) Appearance of first droplet, Min</li><li>b) Completion of melting, Min</li></ul>	175°C 178°C	A-3 of IS 6918 : 1972
ii)	Residue on 150 microns IS sieve, percent by mass, <i>Max</i>	0.5	A-4 of IS 6918 : 1972
iii)	Manganese, ppm, Max	20	A-8 of 18 6918 : 1972
iv)	Copper, ppm, Max	20	A-7 of 1S 6918 : 1972
V)	Loss on drying for 2 h at 105°C, percent by mass, <i>Max</i>	1.0	A-7 of IS 7086 (Part 1): 1973
vi)	Assay (ZDEC content ), percent by mass:		
	a) Unmodified, Min	97.0	Annex A
	b) Surface coated, Min	<b>95</b> .0	do

(*Clauses* 3.2 and 6.1)

#### NOTES

1 In case of dispute in the melting point requirement the final criterion for acceptance shall be the assay requirement.

2 Surface coated/bound materials are likely to contain 2.0 percent surface modifier.

#### ANNEX A

## [ Table 1, Item (vi) ]

### **DETERMINATION OF ASSAY**

#### A-0 CAUTION

During the early stages of test, carbon disulphide is liberated. This is highly inflammable and toxic, and such stages must, therefore, be carried out in a fume cupboard and care must be taken to see that no **naked** flames are near.

#### **A-1 APPARATUS AND REAGENTS**

#### A-1.1 Boric Acid Solution

Make 40 g boric acid (reagent quality) into a paste with water, add one litre hot water and heat until the boric acid has dissolved. Pour the solution into one litre of cold water, mix well, allow to cool.

#### A-1.2 Brilliant Yellow Indicator Paper

A-1.3 Methyl Red Solution --- 0.05 Percent W/W.

**A-1.4 Methylene Blue Solution** — 1.5 Percent W/W.

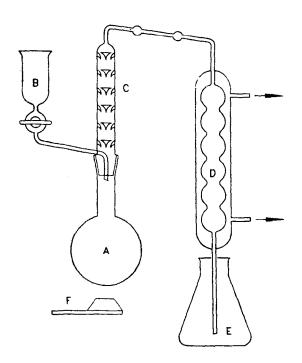
A-1.5 Sodium Hydroxide Solution — 30 Percent W/W.

A-1.6 Standard Solution of Hydrochloric Acid — N/5.

A-1.7 48 Percent W/W Sulphuric Acid Solution

A-1.8 10 Percent Sodium Sulphide Solution W/W

A-1.9 Special Apparatus Required — (see Fig. 1).



- A One litre round bottom flask
- B --- Cylindrical tap funnel
- C Splash head
- D Bulb condenser (with 9 bulbs)
- E 500 ml conical flask
- F -- Electric heating arrangement

#### FIG. 1 APPARATUS ASSEMBLY

It is a round bottom flask of one litre capacity having a standard joint neck fitted with a tap funnel (B), and splash head bulb (C), a vertically placed bulb condenser (9 bulbs) (D), splash head is connected with bulb condenser by the rubber tube. The lower end of the bulb condenser with as short rubber tube (as protector) is inserted inside a 500 ml conical flask (E). F is a bunsen burner, G — is wire gauge over which the flask (A) is held vertically by a strand and clamping device.

#### A-2 PROCEDURE

Weigh accurately 1.5-2.0 g prepared sample into a 250 ml beaker. Add 25 ml of 48 percent sulphuric acid solution and heat on to a steam bath in a fume cupboard for 45-60 minutes. Place some porous chips in the beaker. Cover the beaker with a watch glass, and by means of bunsen burner, maintain the contents of the beaker at a gentle boil, until carbon disulphide is no longer evolved. Continue boiling for 15-30 minutes replacing the water evaporated out at every 5 minutes intervals. Cool the beaker and its contents, wash the watch glass with water, allowing the washings to run into the beaker, and add 50 ml water.

Introduce 50 ml of boric acid solution (see A-1.1) into conical flask E, and insert lower end of the inner tube of the condenser in such a way that it remains below the surface of the boric acid solution.

Put a few porous chips and a brilliant yellow indicator paper inside the flask A.

Transfer the content of the beaker to the flask A through the tap funnel, wash the beaker with 250-300 ml water and transfer them to the flask through the tap funnel, add 200 ml of 30 percent W/W sodium hydroxide solution, and 20 ml of 10 percent sodium sulphide solution. If the solution is not alkaline at this stage, more sodium hydroxide solution must be added till the brilliant yellow paper becomes red.

Heat the content of the flask to boiling and allow the amine to get steam distilled into the conical flask E. Continue heating of the flask A and distillation for one hour till the distillate volume is approximately 250 ml over 50 ml of the boric acid in the conical flask E. Titrate the distillate with N/5 HCl solution.

Carry out a blank steam distillation using 25 ml 48 percent sulphuric acid, a suitable volume of water and same volume of 30 percent sodium hydroxide and sodium sulphide as that used in the test.

Let the net litre of N/5 HCl = A ml

(Net titre = titre of distillates sample — that of blank)

Strength calculated as	
percent zinc dithio	$3.62 \times A$
carbamate (ZDEC) =	mass of the sample
	taken

## ANNEX B

(*Clause* 3.3)

#### METHOD FOR COMPOUNDING AND TESTING OF ZINC DIETHYL DITHIO CARBAMATE (ZDEC)

#### **B-1 TESTING COMPOUND**

As a guidance, the following test compound may be used for testing the performance of Zinc diethyl dithio carbamate (ZDEC) in rubber compound:

Natural rubber Grade A ( conforming to IS 4588 : 1986 )	Parts by Mass 100
Barytes ( conforming to IS 1683 : 1994 )	75
Zinc oxide ( conforming to IS 3399 : 1993 )	10
Stearic acid ( conforming to IS 1675 : 1971 )	1
Sulphur (conforming to IS 8851:1994)	2
Zinc diethyl dithio carbamate (ZDEC)	0- <b>3</b> 5
2 PROCEDURE	

#### **B-2 PROCEDURE**

**B-2.1** Follow the procedure prescribed in NR : 9 of IS 3660.

**B-2.2** The test given below are recommended for each test sample. The approved sample shall also be tested side by side using the same master batch, that is, compound excluding accelerator only.

**B-2.2.1** Determine Mooncy Scorch at 120°C in accordance with the method prescribed in NR : 8 of IS 3660 (Part 7): 1988.

**B-2.2.2** Determine tensile strength modulus at 300 percent elongation at break at different cures 20, 30, 40 and 50 minutes at 141°C (from below to above the expected optimum cure), in accordance with the method prescribed in 1S 3400 (Part 1): 1987.

**B-2.2.3** Hardness on optimum cure at 141°C in accordance with the method prescribed in IS 3400 (Part 2): 1980.

#### **B-3 RESULTS**

The values obtained in the test sample should not vary by more than  $\pm 20$  percent for Mooney scorch and  $\pm 10$  percent for all the other characteristics from those obtained with the approved sample.

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